
**Plastics — Aromatic isocyanates for
use in the production of polyurethanes
— Determination of the isomer ratio
in toluenediisocyanate (TDI)**

*Plastiques — Isocyanates aromatiques pour utilisation dans la
production de polyuréthannes — Détermination du rapport des
isomères dans le diisocyanate de toluène (TDI)*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This third edition cancels and replaces the second edition (ISO 15064:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

- a NOTE has been added to [6.2](#);
- the testing temperature has been added in [Clause 8](#).

Plastics — Aromatic isocyanates for use in the production of polyurethanes — Determination of the isomer ratio in toluenediisocyanate (TDI)

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions prior to use.

1 Scope

This document specifies methods for the measurement of toluene-2,6-diisocyanate in mixtures of the 2,4- and 2,6-isomers. Two methods based on infrared spectroscopy are used to give accurate results over a broad range of isomer concentrations. Method A is applicable to toluenediisocyanate (TDI) samples containing between 5 % and 95 % of the 2,6-isomer. Method B is applicable to TDI samples containing 0 % to 5 % of the 2,6-isomer. Both methods are based on the quantitative measurement of absorption bands arising from out-of-plane C—H deformation vibrations of the aromatic ring at 810 cm⁻¹ and 782 cm⁻¹ (12,3 µm and 13,8 µm).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

<https://standards.iteh.ai/catalog/standards/iso/a22b3/74/-ae40-43e9-ba61-9518a9b89d/iso-15064-2017>

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

isomer

compound having the same percentage composition and molecular mass as another compound, but differing in chemical structure and properties

3.2

isomer content

amount of an *isomer* (3.1) expressed as a mass percentage of the total isomer amount

3.3

polyurethane

polymer prepared by the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

4 Sampling

Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods (for example, sampling an open drum with a thief), even when conducted rapidly, can cause contamination of the sample with insoluble ureas; therefore, blanket the sample with a dry inert gas (e.g. nitrogen, argon or dried air) at all times.

WARNING — Organic isocyanates are hazardous when absorbed through the skin or when the vapours are breathed in. Provide adequate ventilation and wear protective gloves and eyeglasses.

5 Principle

5.1 Method A

In method A (between 5 % and 95 % 2,6-isomer), the infrared spectrum of a cyclohexane solution of the sample is recorded in the 770 cm^{-1} to 840 cm^{-1} (12 μm to 13 μm) region. The absorbance ratio of the 810 cm^{-1} and 782 cm^{-1} bands is measured and converted to % toluene-2,6-diisocyanate from a previously established calibration curve.

5.2 Method B

In method B (0 % to 5 % 2,6-isomer), the absorbance of the 782 cm^{-1} band is measured from an infrared spectrum of an undiluted sample and then converted to % 2,6-isomer from a previously established calibration curve.

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6 Reagents

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Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that the reagents shall conform to the specifications of ISO 6353-2. Other grades may be used, provided that it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.1 Cyclohexane, distilled and stored over silica gel to remove traces of moisture.

6.2 Diisocyanate standards.

Pure samples of 2,4-TDI and 2,6-TDI are required for calibration. The following criteria can be used to judge purity:

for 2,4-TDI: freezing point = 22,0 °C; $n_D^{20} = 1,567\,81$; $\rho_4^{20} = 1,218\,6$;

for 2,6-TDI: freezing point = 18,2 °C; $n_D^{20} = 1,571\,11$; $\rho_4^{20} = 1,227\,0$.

The diisocyanates can be prepared by phosgenating the corresponding pure amines and vacuum-distilling the products. Since these diisocyanates will react with moisture and can discolour in the presence of air, store them under dry nitrogen.

NOTE Gas chromatography (GC) has also been used to establish purity. Isomers determined to be greater than 99,5 % pure by GC with either thermal conductivity (TCD) or flame ionization (FID) detection have been found suitable to be standards.